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INVESTIGATION OF THE DETERMINANTS
OF THE MODULUS OF RUPTURE OF
FUSED SILICA

P. 28.

PART I

February 1, 1961

Document 301

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Contributors to this work:



Number of Pages 22

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I. INTRODUCTION

The report presents a summary of the work that has been done to date on the program presently in progress to determine the effects of grinding method, and surface quality on the modulus of rupture of fused silica. The motivation for such a study as well as the overall objectives are discussed in detail in the document titled "A Proposed Program for Investigating the Determinants of Modulus of Rupture of Fused Silica", July 1960. Figure No. 1 presents in pictorial form the overall procedural flow. This report concerns itself with the results of breaking the group labeled "Set A".

II. PROCEDURES

A. Grinding and Polishing

Sixty fused silica disks, 4" in diameter and 1/4" thick, were numbered for identification and cloth polished on one surface. They were then cemented to blocks in groups of six and the other surface ground and polished in a conventional manner using the following abrasives:

- Blanchard - 150 grit diamond (100 micron particle)
- Fine grind - 2F aluminum oxide (30 micron particle)
- Fine grind - 3F aluminum oxide (20 micron particle)
- Fine grind - KH aluminum oxide (14 micron particle)
- Fine grind - KO aluminum oxide (12 micron particle)
- Polish - Barnesite Rouge

The abrasive sequence was determined from the fine grind and polish schedule followed on other fused silica structural pieces made recently. The overall grinding procedure was uncontrolled in the sense that the amount of material to be removed with each abrasive was left entirely to the discretion of the operator.

A detailed history of abrasive type, material removed in each operation, time of removal, etc., was kept for each sample. All fine grinding and polishing was done by the same optician on the same machine to remove the possibility of operator skill or machine characteristics as an influence on resultant strength.

B. Inspection and Classification

Upon completion of the polishing operation, the samples were cleaned and sent to Quality Control for inspection. The surface area of interest was found to be free from inspectable defects in almost all cases. Controlled surface scratches, therefore, were purposely introduced to approximately 2/3 of the sample pieces, selected at random, and the samples were returned for further inspection. All samples were inspected a minimum of two times by each of two inspectors making a total of four inspections. Samples whose final classification was still in doubt were inspected again by each of the inspectors for a total of six inspections.

Each sample was then placed in one of three groups according to its surface quality within the surface area of interest. The groups are defined as follows:

<u>Group</u>	<u>Scratch</u>
I:	0-60
II:	60-100
III:	100-160

Scratch specifications described in U. S. Government Specification MIL-O-13830 are defined by an arbitrary master at Frankford Arsenal, Pennsylvania and refer to the appearance of all defects which are of a long nature. Duplicates of this master are used for visual side by side comparison with the optical element being tested under the light from a standard 40 watt incandescent lamp.

In case where the proper group classification of a sample was in doubt because of the variation in Q. C. scratch designations, it was placed in the group for which it was originally intended by virtue of the controlled scratch previously introduced.

C. Test Equipment

Figure 2 is a photograph of the equipment used for breaking the test pieces. Load is applied to the sample by the extension of the spring attached to the end of the lever arm. Loading rate can be adjusted to any desired value by varying the voltage to the drive motor. Motor speed is indicated by a tachometer mounted on the motor shaft, and may be kept constant as load increases by fine adjustment of motor voltage.

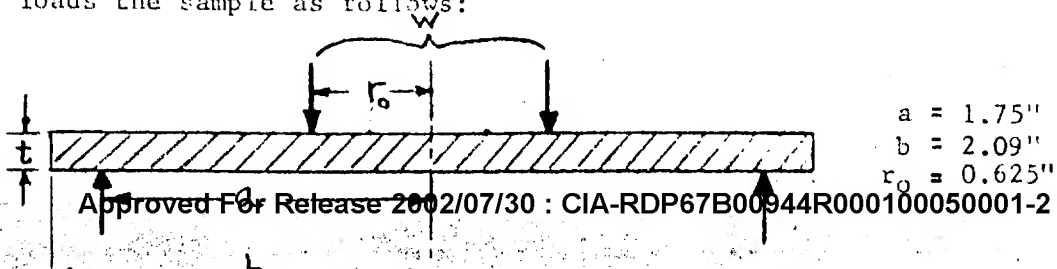
Load measurement is accomplished by means of an Emery hydraulic load cell which transmits a pressure signal to a Honewell pressure transducer. The transducer in turn converts this to an electrical signal which is fed to a pen recorder. Thus a permanent record is made of load versus time for each sample.

Calibration of load cell-transducer assembly was by means of application of known loads measured by a Morehouse ring. Accuracy was found to be within 2% in the test load range.

III. ANALYSIS

A. Breaking Stress

Figure 3 is a photograph of a test sample being broken between the upper and lower rings of the test apparatus. The apparatus loads the sample as follows:



Within the concentric area of radius r_0 , radial and tangential stresses are equal in magnitude, and may be expressed by the equation:¹

$$\sigma_r = \sigma_t = \frac{KW}{t^2 \left(1 + \frac{\pi}{3} K_1 \frac{b-a}{a-r_0}\right)}$$

where K and K_1 are coefficients based on material properties (Poisson's ratio, modulus of elasticity) and test apparatus dimensions (a and r_0). W is total load (lb.).

For the tests under discussion, $K = 0.77$ and $K_1 = 0.32$, the equation may be reduced to:

$$\sigma_r = \sigma_t = \frac{.700W}{t^2}$$

The double ring test is used, rather than the conventional knife-edge method with rectangular samples, because the former produces maximum stresses of equal magnitude throughout the concentric area of radius r_0 , while leaving the edges relatively stress free. The use of the knife-edge method would have resulted in large stresses at the edges of each sample and necessitated the grinding and polishing of the edges of the test samples to the same degree of perfection as the center. Moreover, stress at the scratch would be dependent on exact scratch location and orientation. In the double ring test, however, so long as the scratch is within the concentric area of radius r_0 , the resultant stress at the scratch is independent of scratch location or orientation.

B. Long Time Stress

Figure 4 is a representative curve for the breaking stress of glass at room temperature as a function of time duration.² Shand

¹ Roark, "Formulas for Stress and Strain", McGraw-Hill, 1954, Pg. 194

² Shand, "Fracture Velocity of Glass in Fatigue Range", J. Am. Cer. Soc., January 1961

states that for a particular glass type, the stress concentration factor at the crack tip varies with the one-half power of the crack depth. He further states that with the same initial crack tip stress, the time duration for fracture (stress duration) will vary directly with the initial depth of the crack.

Thus:
$$\frac{a_1}{a_2} = \frac{\sigma_2^2}{\sigma_1^2} = \frac{t_1}{t_2}$$

where a_1 and a_2 = initial crack depths

σ_1 and σ_2 = nominal applied tensile stresses

t_1 and t_2 = time durations to fracture, or stress duration.

Therefore, by selecting any point (σ_1, t_1) on the stress-time curve, and any other point (σ_2, t_2) such that $\frac{\sigma_2^2}{\sigma_1^2} = \frac{t_1}{t_2}$ a line drawn through these two points will represent the locus of equal values of crack tip stress for different magnitudes of initial crack depth. Line A-B on Figure 4 is such a line.

Thus, by shifting the representative curve along line A-B so that it passes through the test point of interest, this single curve may be made applicable to samples having any initial crack depth.

The effective load duration was taken as that required for the final 10% increase before fracture. This relationship is based on actual computations by Shand from tests conducted by him.³

IV. TABULATION OF TEST RESULTS

Table 1 is a tabulation of surface quality inspection results for the test samples. The last column indicates the group in which the sample

³ Shand: Verbal communication, June 1960

was placed as a result of close inspections. It should be noted that in almost all cases where a scratch was present, there was considerable variation between the various Q.C. classifications for a given sample, illustrating the need for a more consistent manner of establishing surface quality.

Table 2 is a tabulation of breaking stress for the test samples arranged by groups. Column 1 shows the actual breaking stress. Column 2 indicates the time in seconds for the load to rise from zero to the breaking load. Column 3 shows the calculated long time stress.

V. DISCUSSION OF DATA

A. Significance of Results

An analysis of variance⁴ performed on the breaking stress data resulted in the conclusion that there was no significant difference between the results of group II and group III. There was, however, definite significance between the results of group I and each of the other groups, and between group I and the composite of groups II and III. Groups II and III, therefore, are treated as a combined sample henceforth in this report.

B. Breaking Stress

Figure 5 is a chart of modulus of rupture versus frequency of occurrence for the test groups.

The data for these distributions are unimodal with averages of 8600 psi for Group I and 7040 psi for Groups II and III and a range of values on either side of the averages of 2000 psi for Group I and 3000 psi for Groups II and III. The distributions were tested to determine their fit with the normal distribution. The plot of the

⁴ Waugh, A.E., "Elements of Statistical Variance", McGraw-Hill, 1943; P236-252

data on probability paper appeared to be representative of a normal distribution. Therefore, the normal distribution parametric prediction techniques were used to determine the lower modules of rupture values.

C. Long Time Stress

Figure 6 shows the frequency of occurrence of calculated long time stress. Introductions of time as a parameter in the calculations of the long time stress has tended to weight the values. When plotted on probability paper, the data appears to be normally distributed. Group I shows a mean value of 3000 psi while the mean for the composite of groups II and III is 2250 psi.

D. Lower Limit Stress Values

We may compute lower limit stress values with varying degrees of reliability according to the formula

$$LV = \bar{X} - tS$$

where LV = lower limit stress value, psi

\bar{X} = sample mean stress, psi

t = a device for computing the lower limit

S = standard deviation

Values of t were obtained from tables prepared by Lieberman⁵ for various reliabilities and percentages of the distribution below the lower limit value. Reliability is the assurance afforded by the formula that the lower limit value computed will be exceeded that percentage of the time. The t values are shown in Table 3 and the lower limit values for breaking stress and long time stress are shown in Tables 4 and 5 respectively. The data of the tables

⁵ G. J. Lieberman, "Tables for One-Sided Statistical Tolerance Limits", Ind. Qual. Control, April 1958

are presented in graphical form in Figures 7 and 8. In both figures, the lower limits for the breaking stress are shown in comparison with the similar values for the long time stress. For example, there is 95% assurance that a value of 5180 psi or less will cause rupture 1% of the time in quartz with a 60 scratch or less. This is equivalent to a 1225 psi long time stress. In quartz with greater than a 60 scratch, the breaking stress is 3370 psi and the long time stress 580 psi for the same reliability and percent of the distribution below the lower limit.

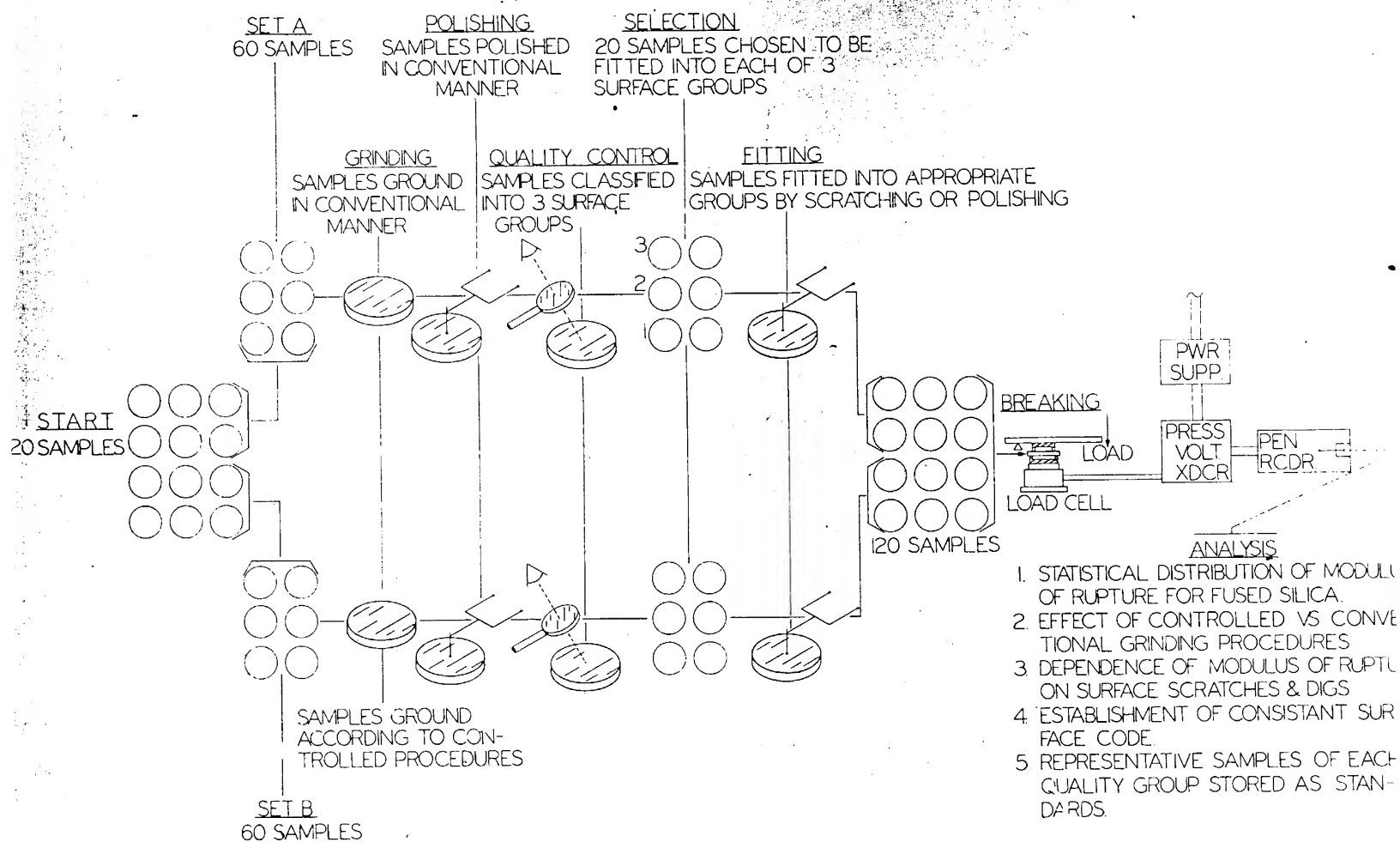
VI. SUMMARY

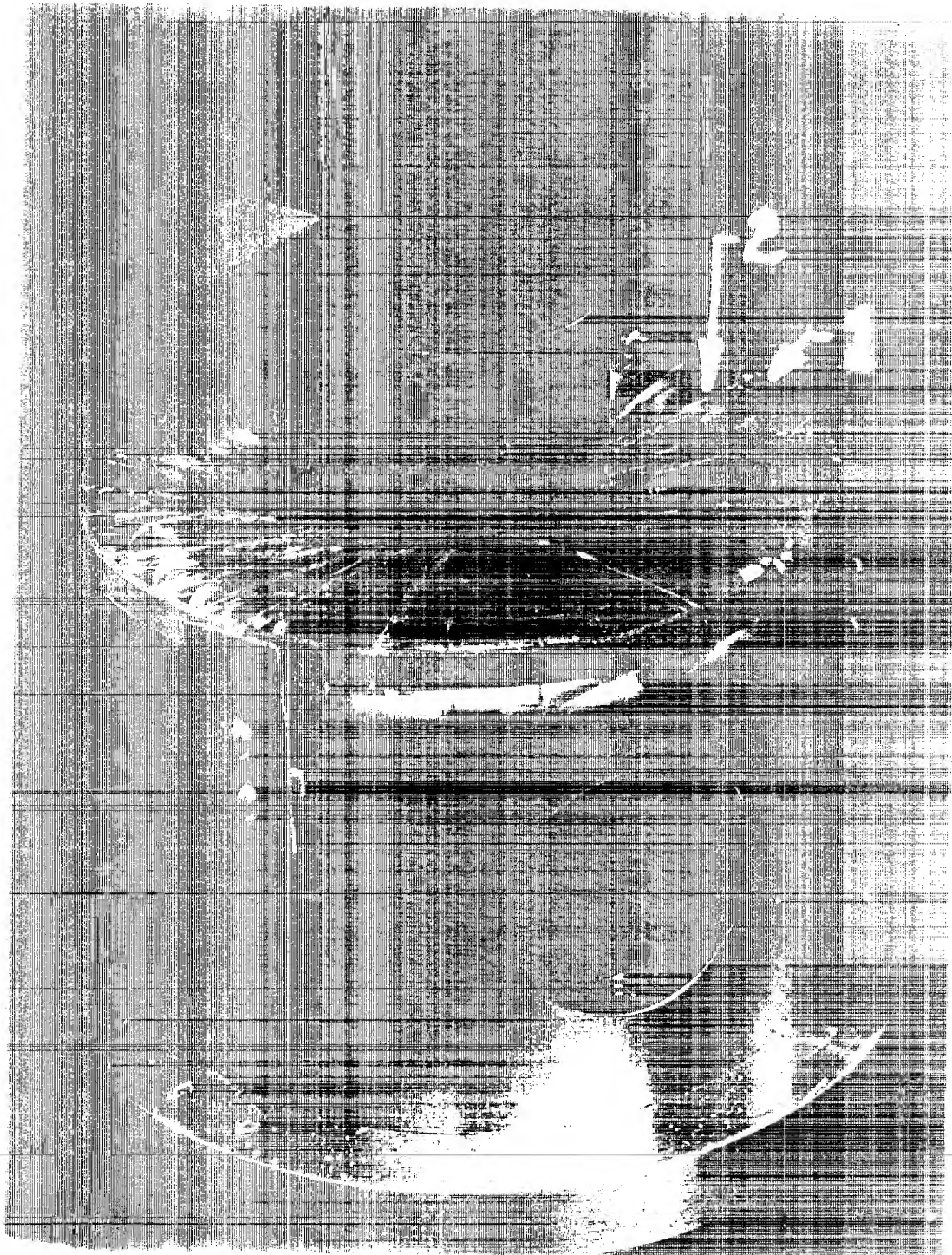
The modulus of rupture for quartz ground and polished according to our present technique was computed for samples of different surface quality. The effect of fatigue on the modulus of rupture was calculated. It was found that the data from samples having a surface quality equal to or better than a 60 scratch was significantly different from those with a surface quality of 30 scratch or worse.

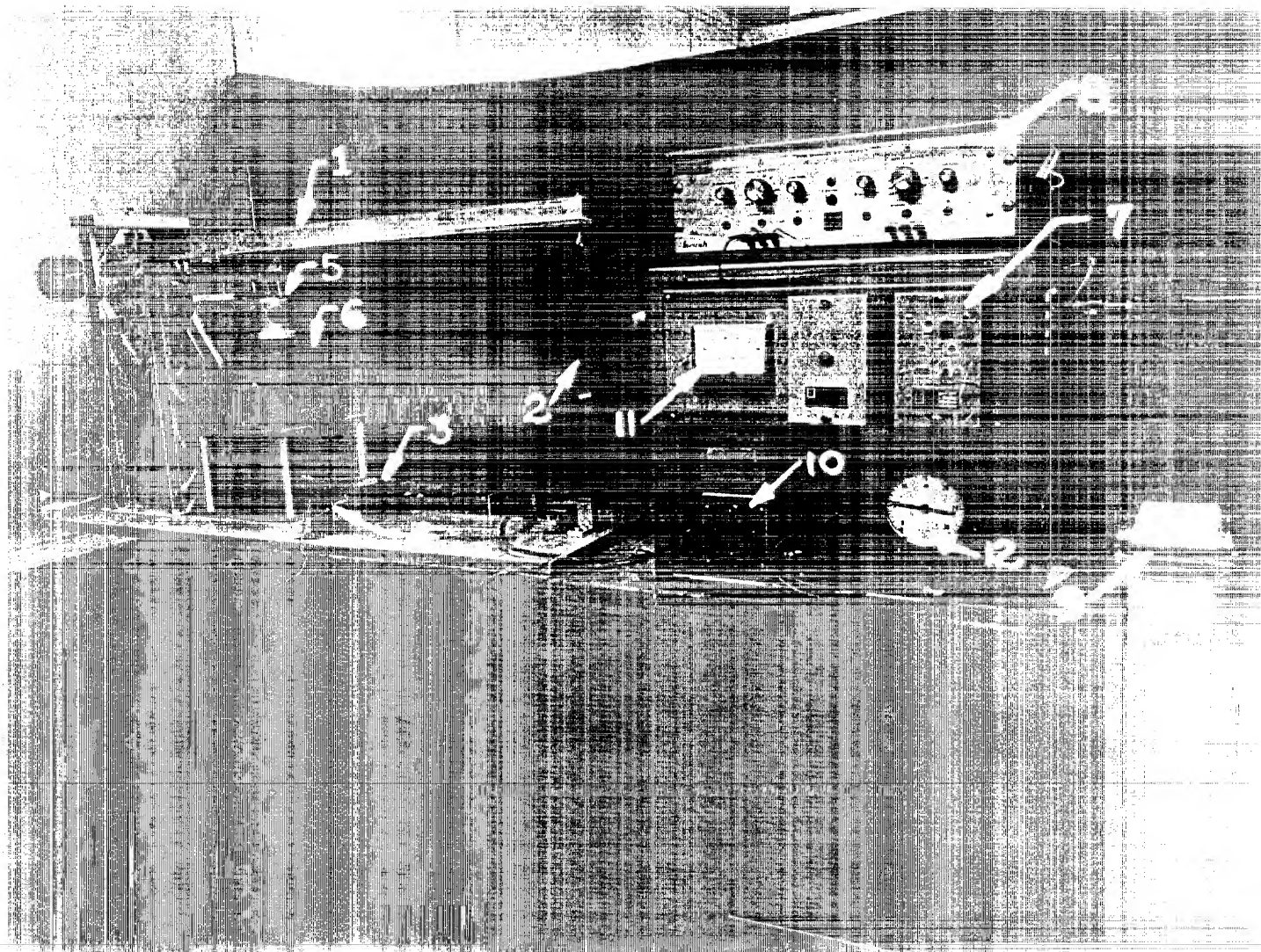
With 99% reliability it was found that only once in a hundred times could we expect failure in the quartz to occur below 4650 psi if its surface quality was better than a 60 scratch, or below 2970 psi if its surface quality was an 80 size scratch or poorer. The effect of fatigue is to reduce these values to 350 psi and 400 psi respectively. It should be noted again that these are calculated values, and that the effect of fatigue on modulus of rupture has not been determined experimentally thus far in this program.

Future plans call for the testing of another set of samples, this time ground and polished according to controlled procedures designed to minimize residual cracks remaining beneath the surface of the quartz as a result of processing. In addition, the effect of fatigue on the modulus

of rupture will be determined experimentally in an attempt to verify the long time stress calculated in this report.







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FIGURE 4

STRESS-TIME CURVE OF FRACTURE

(FRACTURE VELOCITY OF ALUMINUM IN FATIGUE RANGE; E.B. SHAND, J. AM. CER. SOC., JAN '61)

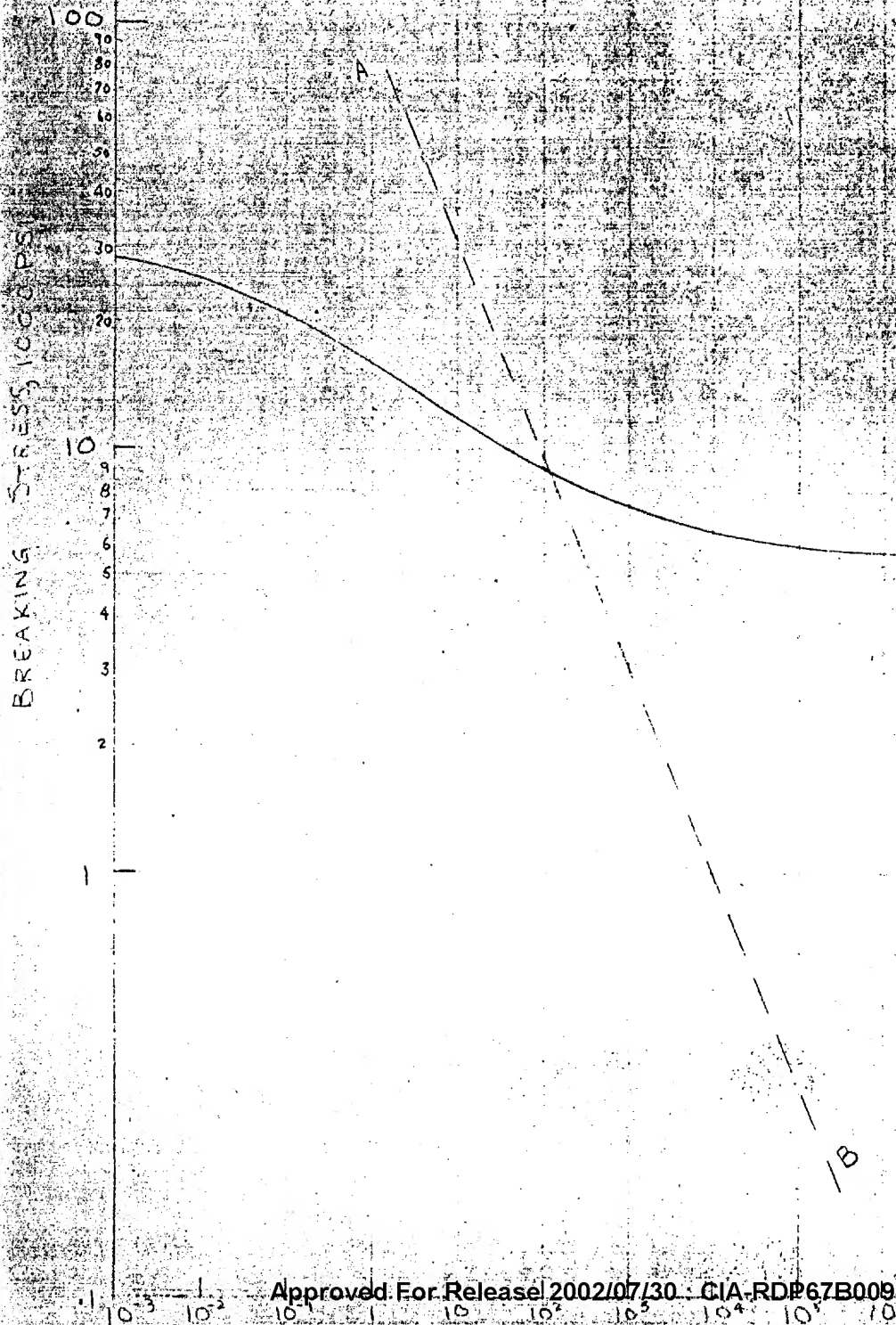


TABLE I

SUMMARY OF SURFACE QUALITY INSPECTION RESULTS

DATE INSPECTOR PIECE NO.	10/3/60 A	10/5/60 B	10/13/60 A	10/14/60 B	10/18/60 A	10/18/60 B	GROUP
1	160	100	100	100	80	80	II
2	0	0	0	0			I
3	0	0	0	0			I
4	0	0	0	0			I
5	0	0	0	0			I
6	0	0	0	0			I
7	0	0	0	0			I
8	0	60	0	0			I
9	80	100	120	100	80	80	II
10	80	80	80	80			II
11	0	0	0	0			I
12	80	80	80	100			II
13	100	100	160	120	160	120	III
14	80	100	100	100			II
15	80	100	100	160	160	160	III
16	160	120	100	100	160	160	III
17	80	100	80	100			II
18	100	100	80	100			II
19	80	100	100	100			II
20	80	160	100	120	160	140	III
21	160	100	120	120	160	160	III
22	100	120	100	100			III
23	80	120	80	80	80	80	II
24	80	80	80	80			II
25	0	0	0	0			I
26	0	0	0	0			I

DATE 10/3/60 10/5/60 10/13/60 10/14/60 10/18/60 10/18/60
 INSPECTOR Approved For Release 2002/07/30 : CIA-RDP67B00944R000100050001-2
 A B A B A B

PIECE NO.							GROUP
27	0	0	0	0			I
28	0	0	0	0			I
29	0	0	0	0			I
30	40	60	40	60			I
31	0	0	0	0			I
32	60	100	80	80			II
33	0	0	0	0			I
34	0	0	0	0			I
35	0	0	0	0			I
36	0	0	0	0			I
37	100	80	80	100			II
38	160	120	120	120	100	100	III
39	80	80	80	80			II
40	160	120	120	120			III
41	80	100	80	100			II
42	160	160	80	120	160	160	III
43	160	80	80	120	120	120	III
44	160	120	160	160			III
45	80	100	80	100			II
46	160	100	160	80	80	80	II
47	80	80	100	80			II
48	80	100	80	80			II
49	80	100	100	160	160	120	III
50	100	80	80	80			II
51	0	0	0	0			I
52	60	120	80	100	80	80	II
53	80	100	160	160	100	120	III

DATE 10/3/60 10/5/60 10/13/60 10/14/60 10/18/60 10/18/60
INSPECTOR Approved For Release 2002/07/30 : CIA-RDP67B00944R000100050001-2
PIECE NO. A B A B

PIECE NO.							GROUP
54	80	100	100	100			II
55	80	120	80	120	100	100	II
56	0	0	0	40			I
57	80	120	160	160	160	160	III
58	80	160	160	120	160	160	III
59	100	160	120	160	160	140	III
60	80	100	100	160	160	160	III

\bar{X} = Mean
S = Standard Deviation
N = Sample Size

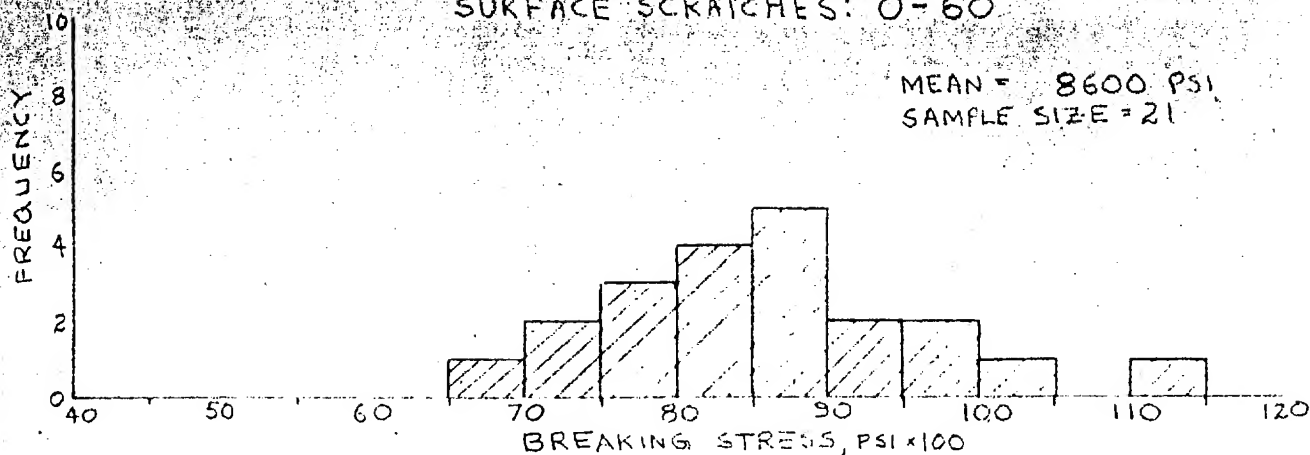
	Breaking Stress, PSI	Long Time Stress, PSI
\bar{x}	6830	2160
s	1235	562
N	37	37

A COMPARISON OF MODULUS OF RUPTURE DATA OF FUSED SILICA
VS. FREQUENCY OF OCCURRENCE FOR SURFACE GROUPS TESTED

GROUP I

SURFACE SCRATCHES: 0-60

MEAN = 8600 PSI
SAMPLE SIZE = 21



GROUPS II & III COMPOSITE

SURFACE SCRATCHES: II: 60-100

III: 100-160

MEAN = 7040 PSI
SAMPLE SIZE = 37

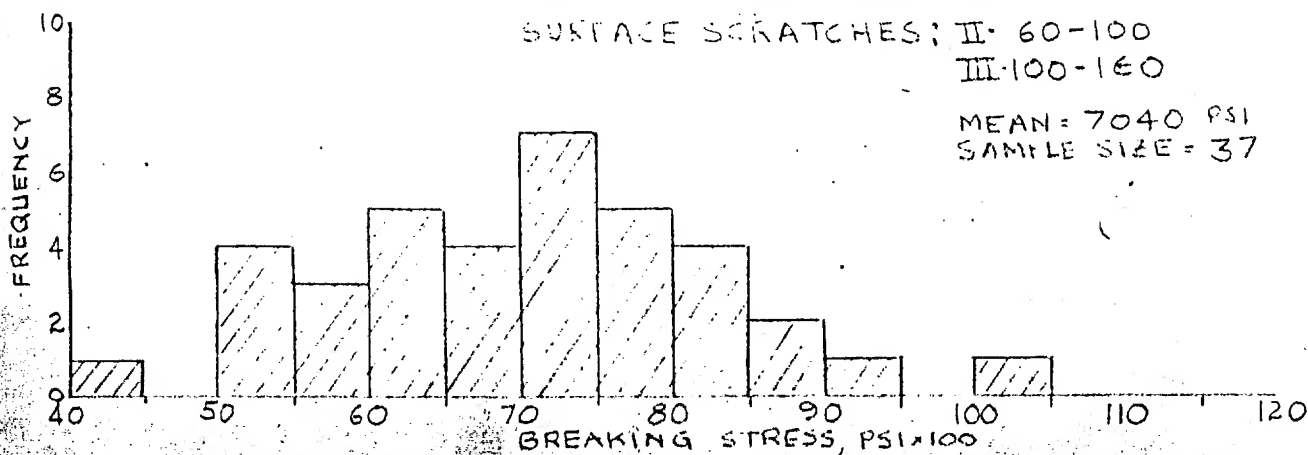
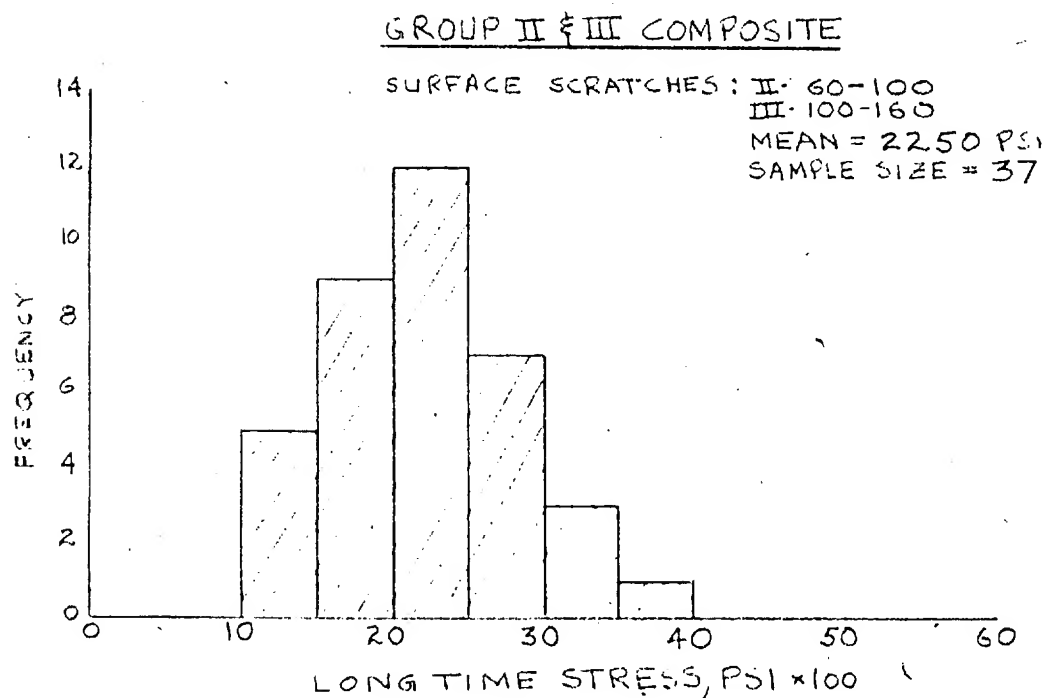
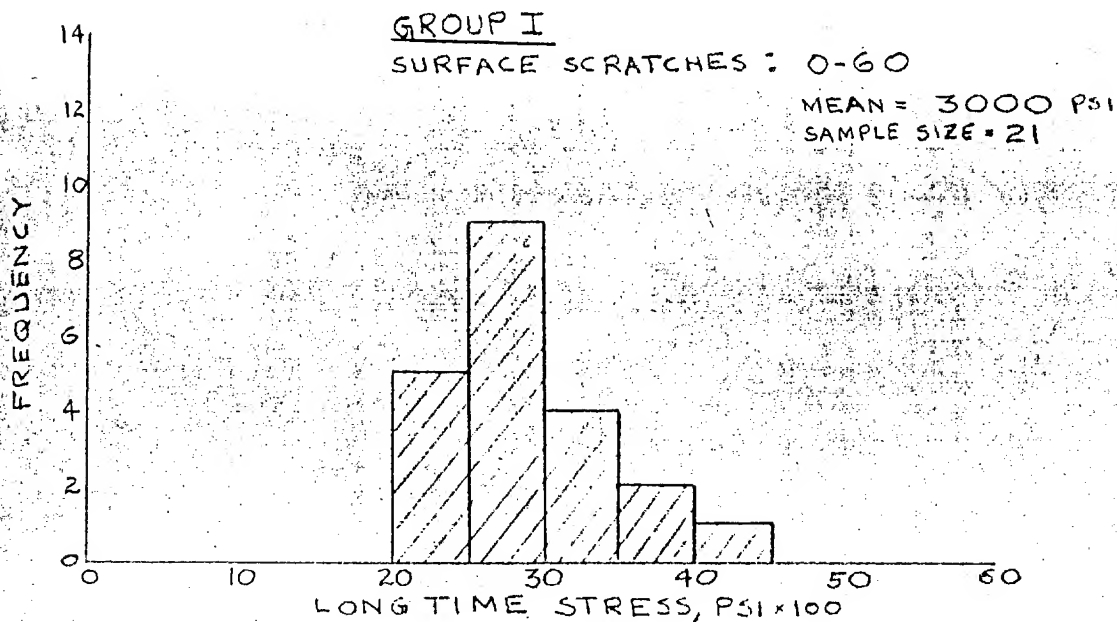


FIGURE 6

CALCULATED LONG TIME BREAKING STRESS OF
FUSED SILICA VS. FREQUENCY OF OCCURANCE



TOLERANCE FACTORS, t FOR VALUES OF α AND γ

RELIABILITY, γ	PCT. OF DISTRIBUTION BELOW THE LOWER LIMIT, α							
	SAMPLE SIZE = 21 (GROUP I)				SAMPLE SIZE = 37 (GROUP II & III)			
	10	5	1	0.1	10	5	1	0.1
.90	1.750	2.190	3.028	3.979	1.613	2.029	2.817	3.710
.95	1.905	2.371	3.262	4.276	1.718	2.150	2.973	3.907
.99	2.241	2.768	3.776	4.932	1.935	2.405	3.300	4.321

TABLE 4

LOWER LIMIT VALUES FOR BREAKING STRESS, PSI

$LV = \bar{X} - ts$

RELIABILITY, γ	PCT. OF DISTRIBUTION BELOW THE LOWER LIMIT, α							
	GROUP I, $\bar{X} = 8600$, $S = 1047$				GROUP II & III, $\bar{X} = 7040$, $S = 1235$			
	10	5	1	0.1	10	5	1	0.1
.90	6770	6310	5430	4430	5050	4535	3560	2460
.95	6605	6120	5180	4130	4920	4385	3370	2220
.99	6255	5700	4650	3430	4650	4070	2970	1720

TABLE 5

LOWER LIMIT VALUES FOR LONG TIME STRESS, PSI

$LV = \bar{X} - ts$

RELIABILITY, γ	PCT. OF DISTRIBUTION BELOW THE LOWER LIMIT, α							
	GROUP I, $\bar{X} = 3000$, $S = 543$				GROUP II & III, $\bar{X} = 2250$, $S = 562$			
	10	5	1	0.1	10	5	1	0.1
.90	2050	1810	1350	840	1345	1110	670	165
.95	1965	1710	1225	675	1285	1040	580	50
.99	1780	1495	950	320	1160	950	395	0

FIGURE 7

RELIABILITY : PCT. OF DISTRIBUTION BELOW
LOWER LIMIT - GROUP I

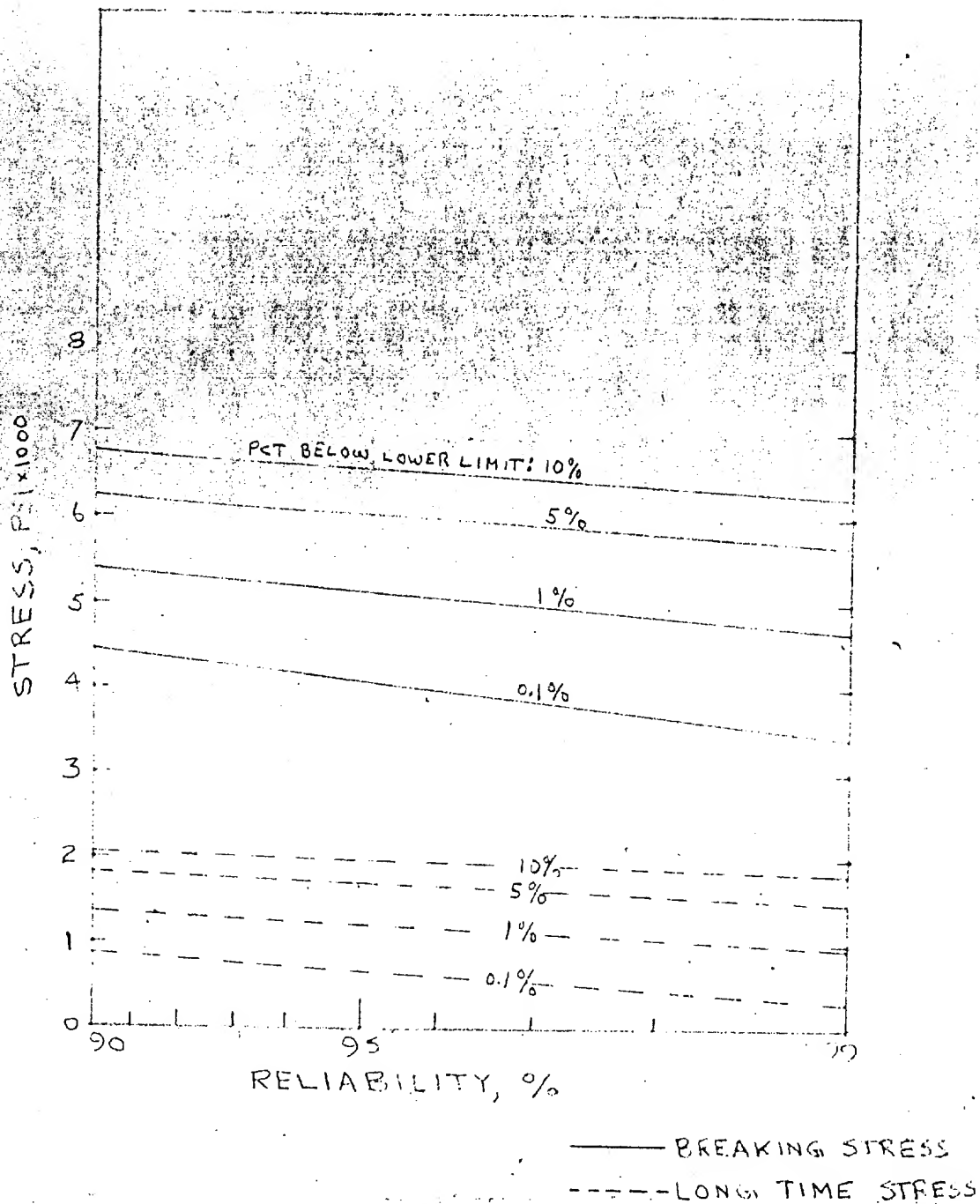
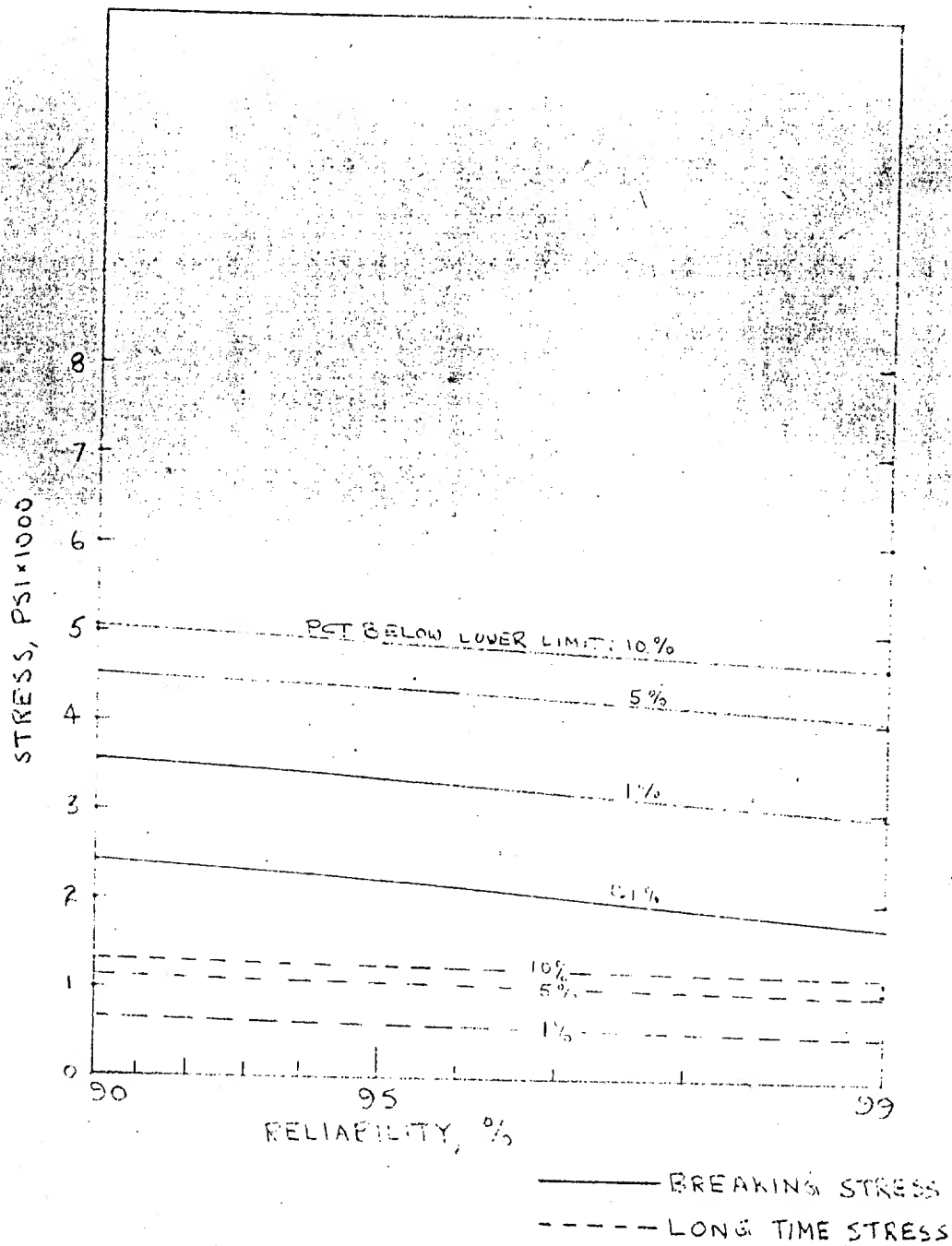


FIGURE 8

RELIABILITY & PCT OF DISTRIBUTION BELOW
LOWER LIMIT - GROUP II & III



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INVESTIGATION OF THE DETERMINANTS
OF THE MODULUS OF RUPTURE OF
FUSED SILICA

PART II


P. 29
February 24, 1961

Document 306

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Contributors to this Work:

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Number of Pages 10

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I. Introduction

This report summarizes the work done during the period 1 February 1961 to 22 February 1961 on the program presently in progress to investigate the determinants of the modulus of rupture of fused silica. Results of previous work as well as general test procedures, method of analysis, and description of test equipment are contained in Document 301, issued 1 February 1961.

Specifically, the work done during this period consisted of determining the modulus of rupture of fused silica, ground and polished according to controlled procedures and comparing the results with those obtained for fused silica ground and polished in a conventional manner. The surface quality of the subject samples (henceforth referred to as set B) was comparable to the Group I samples in Document 301.

II. Controlled Grinding Procedure

A. Theoretical Considerations

Ground and polished glass surfaces, originally milled with a diamond charged wheel, and apparently flawless to microscopic examination may reexhibit the milling pattern on being etched in acid. This phenomenon is the result of vertical fractures, whose sides are in optical contact, extending below the milled surface to a depth which is a function of the size of the grit used in the tool. It has been

determined experimentally by Jones¹ and Preston² that the depths of these fractures is approximately equal to three times the depth of the holes left by chipping in the original milling. Conventional fine grinding techniques make no attempt to insure that these effects have been removed other than an often illusory visual observation that the characteristic milling marks are no longer visible.

B. Test Grinding Procedure

In an attempt to minimize residual sub-surface flaws in the test pieces a fine grinding schedule was specified such that the amount of material removed with each abrasive after milling, was equal to three times the diameter of the average particle size of the preceding abrasive. Specifically the schedule was as follows:

<u>Operation</u>	<u>Abrasive</u>	<u>Avg. Particle Size</u>	<u>Material Removed</u>
Milling	150 grit diamond	.004"	---
Fine grind	2F aluminum oxide	.0012"	.012"
Fine grind	3F aluminum oxide	.0008"	.0036"
Fine grind	KH aluminum oxide	.00055"	.0024"
Fine grind	KO aluminum oxide	.00047"	.0016"
Polish	Barnesite Rouge	---	---

III. Test Results

Table 1 is a tabulation of breaking stress and calculated long

¹F. Shirley Jones, "Latent Milling Marks on Glass," J. Am. Cer. Soc., 29(4), 1946

²F. W. Preston, "The Structure of Abraded Glass Surfaces," Trans. Optical Soc., 23(3) 1921-22

time stress for the test pieces. Of the original sample size of 30, one block of 6 pieces was eliminated due to improper grinding procedure, 3 pieces were eliminated because of size 60 or worse scratches in the central area, and 5 pieces were eliminated because of fracture origin outside the central area. This left a final sample size of 16 to be used for statistical evaluation of results.

Figure 1 shows the distribution of breaking stress and calculated long time stress. It should be noted that the mean values of 12300 psi for breaking stress and 5130 for long time stress represent significant improvement over the corresponding set A mean values of 8600 psi and 3000 psi. As a further matter of interest it might also be noted that of the 21 Group I, Set A samples tested, the highest breaking stress measured (11150 psi) was over 1000 psi less than the mean of 12300 psi for Set B.

The distributions were tested by plotting the data on probability paper and appeared to be representative of normal distributions. Normal distribution parametric prediction techniques, similar to those used for Set A³ were used to determine the lower modulus of rupture values. Table 2 shows the tolerance factors for a sample size of 16. Tables 3 and 4 show the lower limit stress values for breaking stress and long time stress respectively. The data of these tables is presented in graphical form in figure 2. For a detailed description of the use

³Document 301, p. 7

of these tables and curves, the reader is referred to Document 301. Here again the improvement in strength resulting from the controlled grinding procedure is clearly shown. For example, it was found with 99% reliability that only once in a hundred times could we expect failure to occur in the set B samples below 8180 psi whereas for set A, the corresponding value is 4650 psi. Considering the effect of fatigue these values are reduced to 1470 psi and 950 psi respectively.

IV. Verification of Stress Calculations

The formulas used for calculating breaking stress were verified by bonding an SR-4 strain gage to an aluminum disk of similar dimensions as our quartz test disks, and comparing the stress values calculated by our formulas with those calculated on the basis of strain gage reading. The results shown in figure 3 appear to be in good agreement with each other, representing an approximate 3% difference which is believed to be due to a calibration error in the millivac instrument used to measure load cell output for this test. It is felt that this error would be negligible in our actual tests, as the pen recorder is always calibrated using a precision potentiometer prior to each test run.

V. Summary

The modulus of rupture of quartz, ground and polished according to controlled techniques designed to minimize residual sub-surface flaws was determined for samples having a surface quality of 60 size scratch or better. The results were compared with those obtained for samples

of similar surface quality prepared according to conventional grinding techniques. A significant improvement in strength resulted from the use of the controlled technique.

Test samples are presently being prepared for use in the experimental determination of the effect of fatigue on modulus of rupture, in an attempt to verify our long time stress calculations. Our present schedule calls for this study to be completed for conventionally ground (set A) samples by approximately May 1, 1961. In addition, the effect of surface quality on the modulus of rupture of quartz, ground and polished according to controlled techniques will be determined.

TABLE 1SUMMARY OF MODULUS OF RUPTURE CALCULATIONS -SET B
(UNSCRATCHED SURFACE)

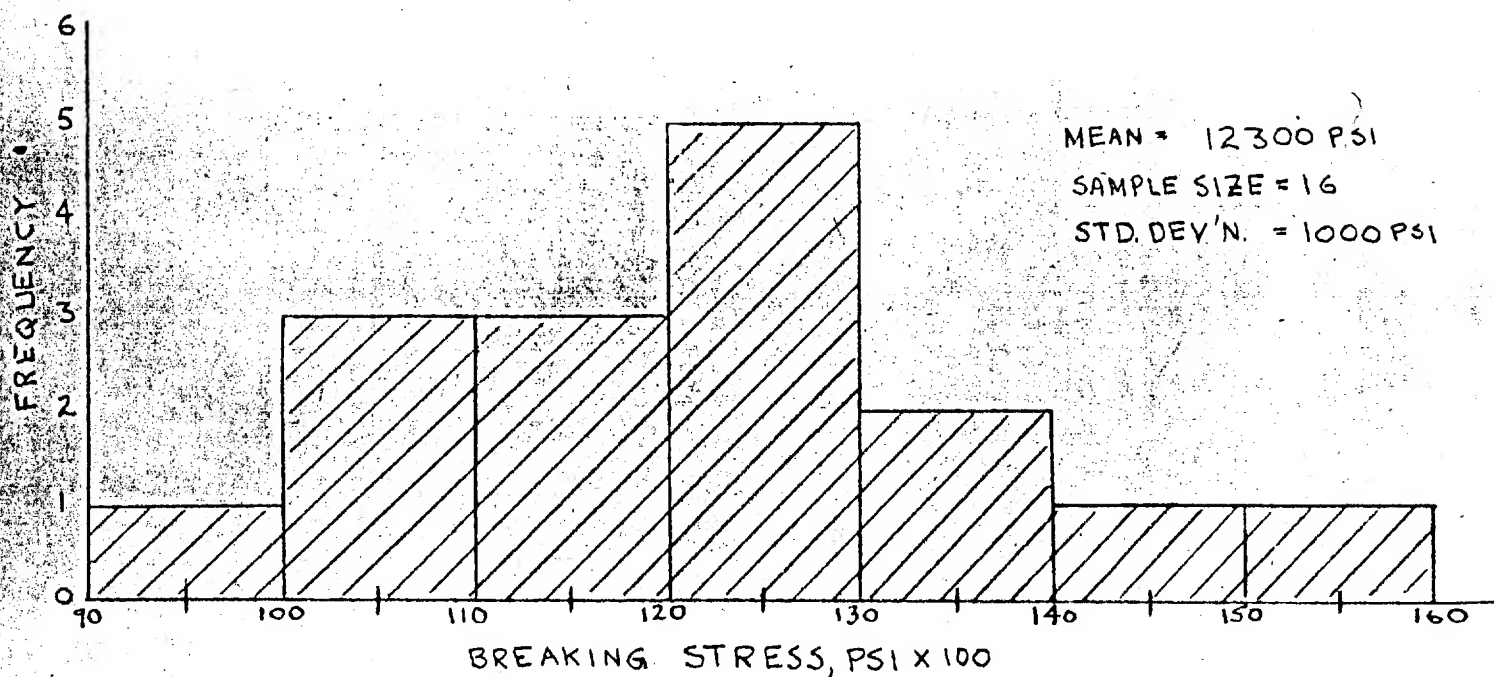
<u>PIECE NO.</u>	<u>BREAKING STRESS, PSI</u>	<u>TIME, SEC</u>	<u>LONG TIME STRESS, PSI</u>
67	14800	46.3	6800
68	13000	43.7	5700
69	13900	44.5	6200
71	15100	46.5	7200
72	12900	41.6	5600
73	11700	35.3	4700
74	10400	34.5	4100
75	11200	35.3	4400
76	11000	37.5	4400
79	12700	40.2	5200
80	10200	29.7	3800
81	12500	38.6	5200
83	11500	34.6	4400
84	9600	28.0	3600
86	12200	35.5	4900
88	13900	36.0	5900
\bar{x}	12300		5130
n	16		16
s	1000		890

NOTE: The following samples were eliminated from test results:

<u>PIECE NO.</u>	<u>REASON</u>
61-66	Improper Grinding Technique
77, 78, 87	Scratch in Central Area
70, 82, 85, 89, 90	Fracture Origin Outside Central Area

FIGURE 1

MODULUS OF RUPTURE OF FUSED SILICA - SET B
(UNSCRATCHED SURFACE)



CALCULATED LONG TIME STRENGTH OF FUSED SILICA - SET B
(UNSCRATCHED SURFACE)

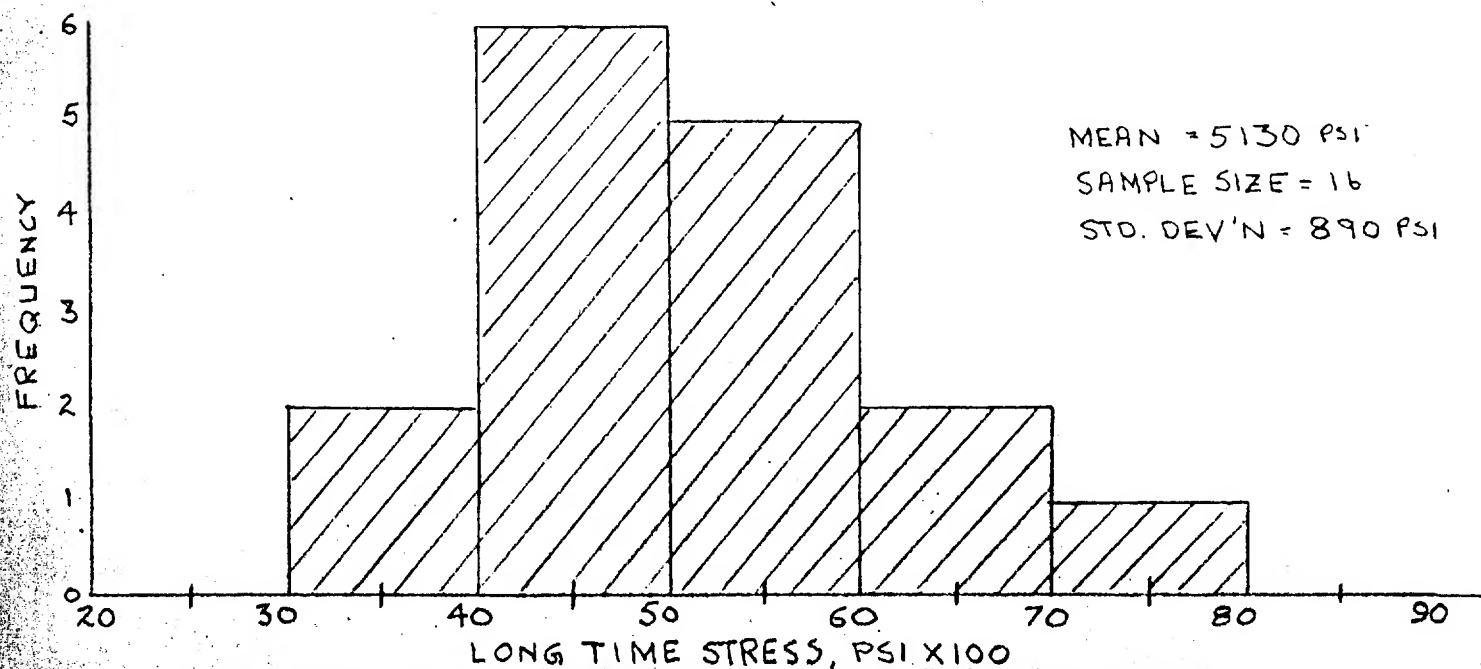


TABLE 2

TOLERANCE FACTORS, t FOR VALUES OF α AND γ

	% DISTRIBUTION BELOW LOWER LIMIT, α			
	SAMPLE SIZE=16			
RELIABILITY, γ	10	5	1	0.1
.90	1.842	2.299	3.172	4.164
.95	2.032	2.523	3.463	4.534
.99	2.458	3.028	4.124	5.374

TABLE 3

LOWER LIMIT VALUES FOR BREAKING STRESS, psi
 $LV = \bar{X} - tS$

	% DISTRIBUTION BELOW LOWER LIMIT, α			
	$\bar{X} = 12,300 \text{ psi}$ $S = 1,000 \text{ psi}$			
RELIABILITY, γ	10	5	1	0.1
.90	10,460	10,000	9,130	8,140
.95	10,270	9,780	8,840	7,770
.99	9,840	9,270	8,180	6,930

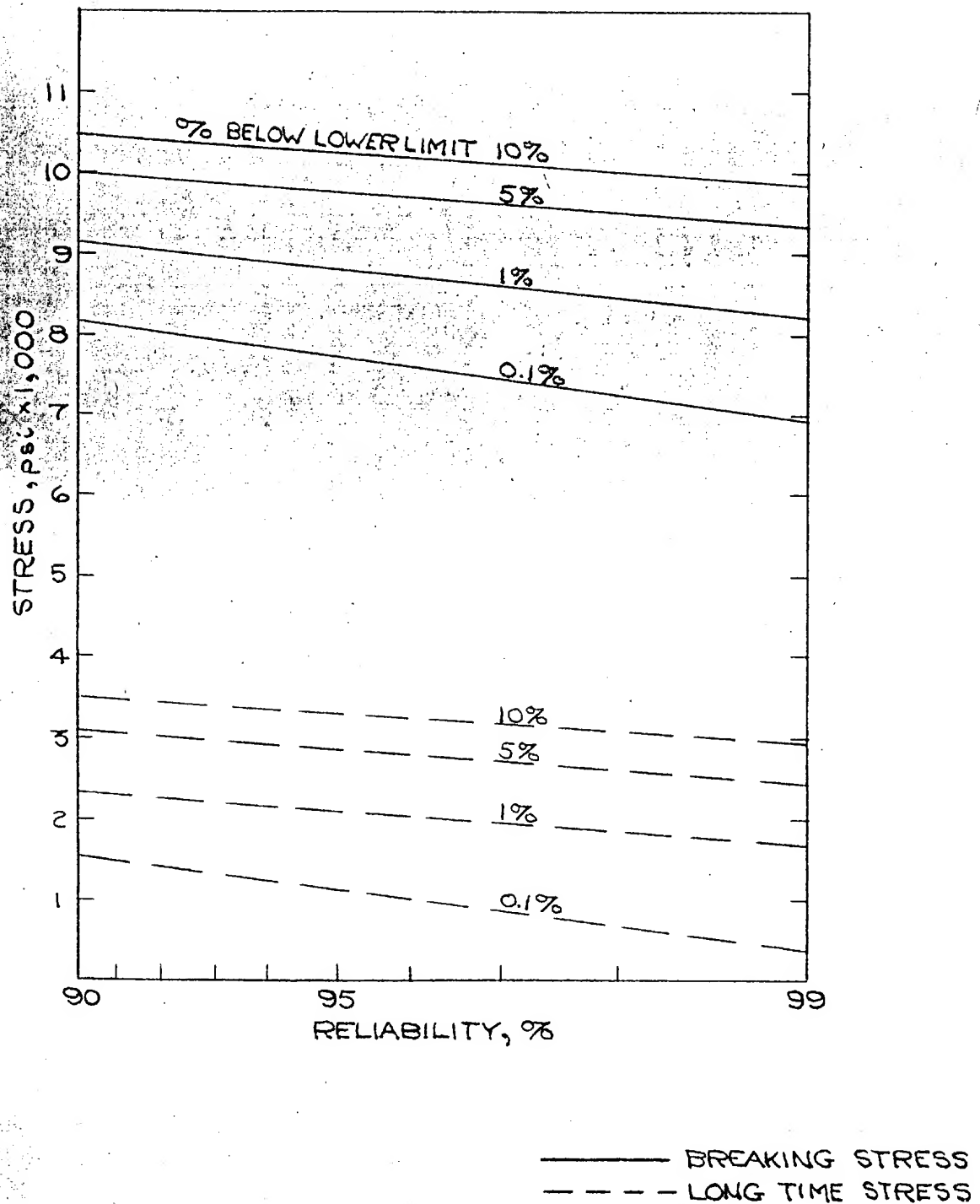
TABLE 4

LOWER LIMIT VALUES FOR LONG TIME STRESS, psi
 $LV = \bar{X} - tS$

	% DISTRIBUTION BELOW LOWER LIMIT, α			
	$\bar{X} = 5,130 \text{ psi}$ $S = 890 \text{ psi}$			
RELIABILITY, γ	10	5	1	0.1
.90	3,490	3,090	2,310	1,430
.95	3,320	2,890	2,050	1,100
.99	2,940	2,430	1,470	350

FIGURE 2

RELIABILITY & % DISTRIBUTION BELOW LOWER LIMIT



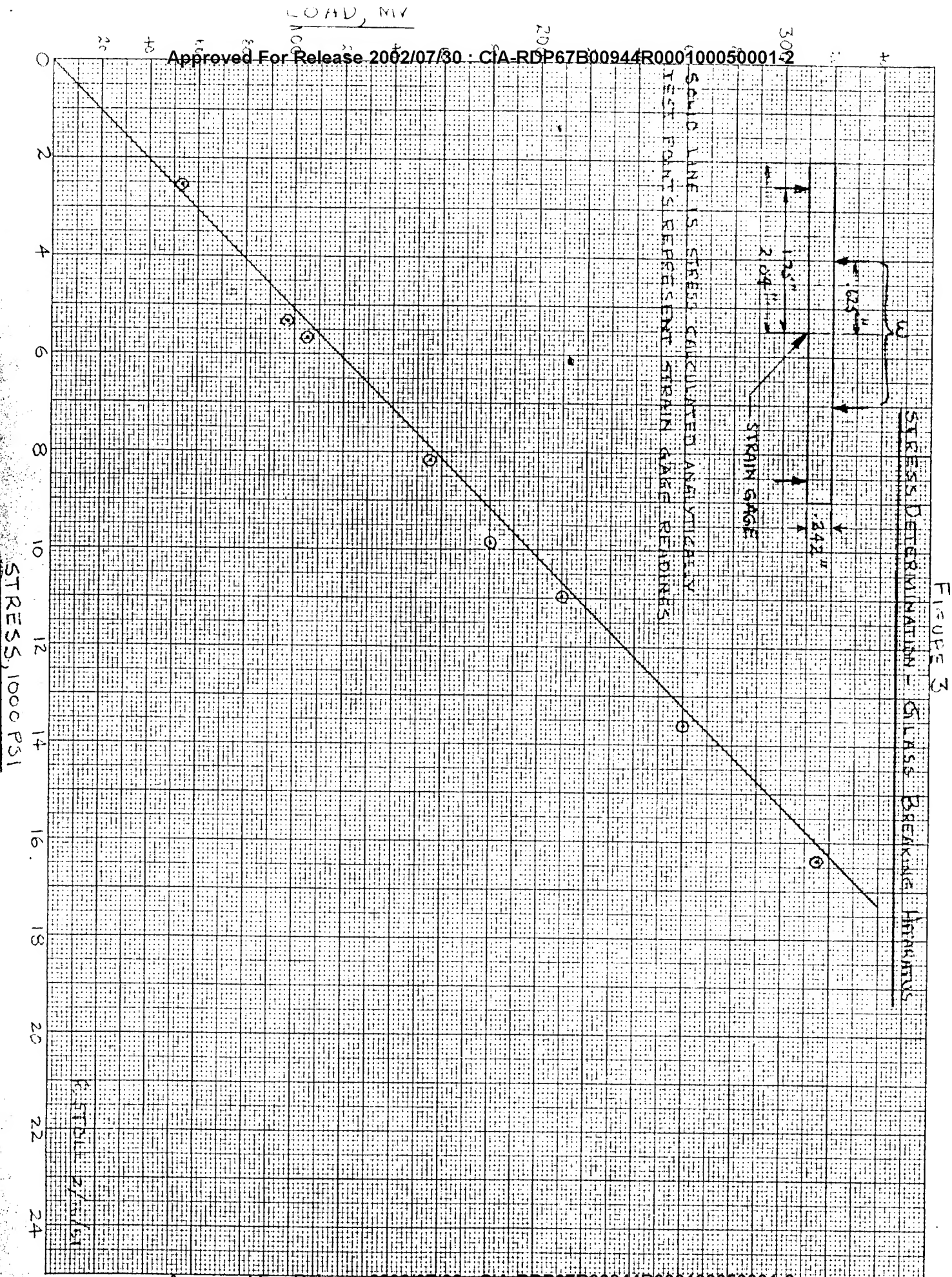


FIGURE 3